2009

Soil sample handling for routine analysis of plant-available soil potassium

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Soil sample handling for routine analysis of plant-available soil potassium

by

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A thesis submitted to the graduate faculty

in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Major: Soil Science (Soil Fertility)

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Ames, Iowa

2009

To:

My wife Kelly

My son Owen

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1: GENERAL INTRODUCTION

Soil testing is an important diagnostic tool to assess nutrient sufficiency of plants. The plant-availability of soil potassium (K) has been particularly difficult to assess from a soil testing perspective. Most of the K in the soil is unavailable to the plant, so any soil test to predict sufficiency for plants must extract a fraction of K that is available or is proportionally related to what will be available. Both soluble and exchangeable soil K have been considered as plant available fractions of K. Over many decades, many different extracting solutions and extraction procedures have been used to estimate these K fractions. The most widely used soil-test method in Iowa, the U.S., and the world involves K extraction with an ammonium-acetate solution (NH_4AOc). Until the early 1960's, this test was used in Iowa and the rest of the world on air-dried or oven-dried samples. From the middle 1960's until the late 1980's, the Iowa State University Soil Testing Laboratory used a field-moist method for this test. This change was decided because at the time Iowa research showed that extracting K from field-moist samples was a better predictor of K sufficiency than extracting K from air-dried or oven-dried samples. No other public or private laboratory adopted this field-moist test, however, because it was considered impractical for routine soil testing. Threfore, in 1988 the laboratory procedure and soil-test K interpretations were changed to an oven-dried soil sample method.

Iowa field research and farmers' observations during the 1990's and early 2000's showed increasing frequency of K deficiency symptoms in corn and soybean. These symptoms were observed in low-testing soils, but also in soils that tested at or near optimum levels according to the soil-test K interpretations at the time. Therefore, new field calibration

research results for corn and soybean for the NH4OAc method and other methods (such as the Mehlich-3 K test) based on oven-dried samples were used to update soil-test K interpretations in 2002. Large response variation across soils with similar soil-test K levels was still observed for both corn and soybean crops when oven-dried K tests were used, however, which indicated a need for continuing research to improve soil testing methods and interpretations for K in Iowa soils.

The goal of this research was to expand recent work in Iowa by further studying K extraction methods and its use to evaluate soil K sufficiency for crops. Specific objectives were to compare soil-test results and relationships between soil-test K and crop yield response to K fertilization when soil K was measured with the NH4OAc extractant from field-moist samples, oven-dried soil samples, and also after applying different laboratory treatments to oven-dried samples in an attempt to revert the effect drying has on the K release.

THESIS ORGANIZATION

This thesis is presented as one paper suitable for publication in scientific journals of the American Society of Agronomy or Soil Science Society of America. The paper is entitled "Soil Sample Handling for Routine Analysis of Plant-Available Soil Potassium." The paper includes sections for an abstract, introduction, materials and methods, results and discussion, conclusions, references, tables, and figures. The paper is preceded by a general introduction and is followed by a general conclusions section.

CHAPTER 2: SOIL SAMPLE HANDLING FOR ROUTINE ANALYSIS OF PLANT-AVAILABLE SOIL POTASSIUM

A paper to be submitted to American Society of Agronomy or Soil Science Society of America Journal

Brian E. Hill and Antonio P. Mallarino

ABSTRACT

Soil K extraction with neutral 1 *M* ammonium-acetate (NH_4OAc) based on air-dried or oven-dried samples is the most widely used soil test for K. It has long been recognized that sample drying often increases K extracted by this test. An NH_4OAc K test based on field-moist samples (MK) was used until 1988 by the Iowa State University laboratory but was abandoned because no private laboratory adopted it. In the 1960's work was done to look at different chemical treatments that prevent K from being released upon sample drying or revert the drying effect before K extraction. The objectives of this study were to assess the impact of sample drying and two post-drying treatments on extracted soil K and its relationships with crop yield response to K fertilizer. Sample treatments before measuring K with the NH_4OAc extractant were no treatment (test on field-moist samples), drying at 40 C (DK), water incubation (Rewet K), and water-octanol incubation (Octanol K). Soil samples were collected from single-year and multi-year K response trials with corn (*Zea mays* L.) or soybean [*Glycine max* (L.) Merr.] that were conducted at 19 Iowa fields from 2001 to 2004. Soil K extracted with DK was higher than for MK, and these tests were linearly correlated (r^2

= 0.53). The K extracted with Rewet-K and Octanol-K tests often was less than for DK, and Octanol-K values and were as low as values for MK. Rewet-K and MK-values were linearly related (r^2 = 0.59). Octanol-K was linearly but poorly related to MK (r^2 = 0.35). The difference between K extracted by MK and that extracted by DK, Rewet K, or Octanol K tended to be inversely related to the soil K level. Soil types and measured soil properties (texture, cation exchange capacity, pH, and organic matter) did not clearly explain the differences between K tests. The Octanol-K test did not measure plant-available appropriately and was the worst method for prediction of crop response to K fertilization. The Rewet K test was approximately similar to DK and MK and would be more practical for routine testing than MK test. Further field calibration research including a wider range of field conditions is needed to better compare these three K testing methods.

Abbreviations: CN, Cate-Nelson; DK, oven-dried K test; LP, linear-plateau; MK, field-moist K test; NH_4OAc, ammonium-acetate; Octanol K, K extracted after water-octanol incubation; QP; quadratic-plateau; Rewet-K, K extracted after incubation with water.

INTRODUCTION

Potassium is the seventh most abundant mineral in the Earth's crust. Of the 13 essential minerals, only N is absorbed more by plants. The amount of K in soils is greater than the plant needs, but not all is available for uptake. Soil K can be divided into four fractions. Soluble K and exchangeable K being available to the plant for uptake, but these fractions are in relatively small amounts compared to fixed K or non-exchangeable K and to structural K, which need to undergo chemical or physical change to become available. When the plant K uptake becomes greater than the K supply from soluble and exchangeable K fractions the K supply from the non-exchangeable pool may be insufficient and, therefore, plant growth and yield may be limited and deficiency symptoms may occur.

Soil testing is an important diagnostic tool in crop production systems to assess plant K availability. An estimation of exchangeable soil K by extracting soil with neutral 1.0 *M* NH_4OAc from air-dried or oven-dried soil samples is the most widely used soil test for K and it provides the basis for K fertilizer recommendations. The Mehlich-3 extractant, for which adoption by soil-testing laboratories is increasing, includes NH_4OAc in its extracting solution. These two tests are the suggested soil K tests for soils of the north-central region of the USA by the North-Central Regional Committee for Soil Testing and Plant Analysis (Warncke and Brown, 1998). It has been stated that a soil should be tested with minimal chemical or mechanical disturbance or alteration in the process of sample preparation to better represent field conditions (Gelderman and Mallarino, 1998). However testing *in situ* is not technically feasible yet, and soil samples are usually dried and crushed to simplify sample handling and provide a homogenous mix for subsampling. It has long been

recognized that drying soil samples can influence the amount of K extracted with the NH_4OAc test. Many researchers have found an increase on extractable K upon drying (Steenkamp, 1928; Atoe, 1947; Luebs et al., 1956; Hanway and Scott, 1959) while others reported that soils high in exchangeable K tend to fix K and soils low in exchangeable K tend to release K upon sample drying (Cook and Hutcheson, 1960; Hanway et al., 1962; Haby et al., 1988).

The impact of sample drying on changes in soil exchangeable K depends on the equilibrium concentration of K, the deviation from the equilibrium concentration at sampling time, and soil mineralogy (Haby et al., 1990). Cook and Hutchenson (1960) postulated an equilibrium level of 196 mg K kg^{-1} for Kentucky soils and indicated that K is fixed upon drying when exchangeable K is higher than that level for field-moist samples and released when the K level is lower. Dowdy and Hutcheson (1963), also working with selected Kentucky soils, reported that the field-moist K equilibrium value of these soils was 175 mg kg^{-1}. Haby et al. (1988) found that the field-moist K equilibrium value of 18 Montana soils was 420 mg kg^{-1}. These authors stated that clay mineralogy of the soils was closely related with K release or fixation observed upon soil sample drying. They found that illite appeared to be the source of K released by drying, while vermiculite and montmorillonite were associated with K fixation. McLean and Watson (1985) stated that, if the soil is relatively low in exchangeable K, soil sample drying causes scrolling of layers of the micaceous clay that releases non-exchangeable K. However, if the soil is relatively high in exchangeable K or has had K added to it recently, drying usually drives water out from between the layers, causing them to collapse and trap K in a non-exchangeable form (McLean and Watson,

1985). Depending on the relative magnitude of these two mechanisms a net release, fixation, or no change in exchangeable K could be observed.

Luebs et al. (1956) in a greenhouse study with 13 Iowa soils showed that K extracted from field-moist samples was better correlated with corn plant uptake than K extracted from air-dried soil. Superior correlations with field-moist samples were also found in K studies conducted in the north central region in greenhouse with millet (Barber et al., 1961), in the field with alfalfa (Hanway et al., 1961), and with corn (Hanway et al., 1962). The improved relationship resulting from undried samples was attributed generally to a variable increase in exchangeable K when soils were dried (Grimes and Hanway, 1967). Because of the effect of sample drying on K test results and the allegedly better correlation with plant uptake found for a test of undried samples, a method for testing field-moist soil samples based on a slurry was developed and implemented in Iowa in the late 1960s, and it was among tests suggested by the North-Central Region NCR-13 soil testing committee (Brown and Warncke, 1988; Eik and Gelderman, 1988). Field correlations for corn and soybean for this slurry K test from long-term Iowa experiments were published by Mallarino et al. (1991a, 1991b). The Iowa State University Soil and Plant Analysis Laboratory discontinued analyzing samples with the slurry K test in 1988, however, because no private laboratory adopted it due to impractical laboratory procedures (mainly soil moisture determination and slurry preparation). Based on comparisons of amounts of soil K extracted using dried (35 to 40 ºC) or moist soil samples (not field calibrations) the soil-test interpretation categories for the slurry NH_4OAc K test were increased by a factor of 1.25 for Iowa recommendations published in 1988 (Voss and Killorn, 1988) and 1996 (Voss et al., 1996). The old database for the slurry K test and a 1.25

factor continued to be used for the NH_4OAc K test and the Mehlich-3 K test (for the first time, although interpretations were similar for both tests) for updated recommendations published in 1999 (Voss et al., 1999).

Increasing frequency of K deficiency symptoms in corn and soybean was observed across Iowa in the 1990's. These symptoms occurred in low-testing soils, and also in soils that tested medium or optimum according to the soil-test K interpretations at the time (Voss et al., 1996, 1999). Therefore, new field calibration research results for corn and soybean for NH_4OAc and the Mehlich-3 K tests based on oven-dried samples (Mallarino et al., 2002) were used to update soil-test K interpretations (Sawyer et al., 2002). However large response variation across soils with similar soil-test K levels was still observed for both corn and soybean crops when air-dried or oven-dried K tests were used (Mallarino and Blackmer, 1994; Mallarino et al., 2002, 2003), which indicated a need for continuing research to improve soil testing methods and interpretations for K in Iowa soils. Voss (1998) based on a national survey of recommendations by land grant universities strongly suggested a need to update the soil-test calibration data supporting recommendations for many regions, although soil-test calibration research has become difficult due to unwillingness of traditional agricultural research funding sources to fund this kind of research (Beegle, 2005).

Recent Iowa research (Barbagelata et al., 2005) developed field calibrations for the NH_4OAc soil K test based on field-moist and dried samples for corn and soybean. For moist soil samples of this research, the authors maintained most procedures for the old field-most test except that carefully sieved moist samples to be extracted instead of using a slurry as before, but correlated the old and new method for about 100 samples. The results showed

that the two methods extracted comparable amounts of K and, as the old method, the new method extracted less K and provided better correlations with crop yield response than analysis of air-dried samples. Returning to a field-moist method for K analysis will face the same hurdles of adoption by other laboratories it did in the 1980's. Decades ago, Scott and Bates (1964, 1965) studied soil samples with or without several organic compounds on amounts of K extracted. In follow-up studies (Bates and Scott, 1969) evaluated a method of K reversion that involved treating air-dried and crushed samples with water and n-octanol for 48hours at 110 C. Correlation coefficients between field-moist exchangeable K and exchangeable K determined by air-dried, air-dried rewet, and air-dried and octanol-treated samples were 0.03, 0.09, and 0.42 respectively. Correlation coefficients between K uptake by millet in a greenhouse study and exchangeable K determined by field-moist, air-dried, air-dried Rewet, and air-dried octanol-treated soil where 0.89, 0.03, 0.11, and 0.44, respectively. The reversion technique has good potential to allow laboratories to prepare samples by air-drying to maintain a high throughput and a homogenous mix and improve prediction of K sufficiency for crops compared with direct extraction from dried samples. However, this K reversion procedure has not been compared with other procedures on the basis of field calibration research. Therefore, the objectives of this study was to assess effectiveness of the NH4OAc test based on reversion by rewetting and n-octanol treatments on oven-dried samples for corn and soybean in Iowa soils by conducting a field calibration study.

METHODS AND MATERIALS

This study used soil samples and grain yields from K response field trials with corn and soybean conducted in Iowa from 2001 to 2004. Data was selected from sites that encompassed a wide range of yield responses and soil-test K values measured with the NH4OAc test on field-moist and oven-dried soil samples as summarized by Barbagelata et al. (2005). The chosen soil samples and yield data were from eleven single-year trials, five two-year trials, and three, three-year trials (19 fields). Therefore, there were a total of 30 trial-years, 17 with corn and 13 with soybean. All trials used a traditional strip-trial methodology adapted to use precision agriculture technologies such as global positioning systems (GPS), grain yield monitors, and geographical information systems (GIS). Each trial evaluated two treatments, which consisted of a control receiving no K and a single K rate that ranged from 100 to 186 kg K ha^{-1} depending on the field. The K fertilizer (commercial KCl) was broadcast for corn or soybean with commercial fertilizer spreaders to strips measuring 18.3 m in width in all fields and 250 to 450 m in length depending on the field. In multi-year trials, the K treatments were re-applied each year for corn-soybean or soybean-corn crop sequences. The K treatments and three to four replications were arranged in randomized complete-block designs. Nitrogen and P fertilizers were applied uniformly across all strips of each trial by the cooperating farmers using rates as high or higher than the rates recommended by Iowa State University (Sawyer et al., 2002; Blackmer et al., 1997). Other crop management practices, such as tillage, corn hybrids, soybean cultivars, seeding dates and rates, and weed control, were those normally used by the farmers.

Composite soil samples (12 cores, 0-15 cm depth) were collected in the fall after harvest of the previous crop and before applying K treatments using a systematic, grid-point sampling method (Wollenhaupt et al., 1994) adjusted to the field design of each trial. For single-year trials or the first-year of multi-year trials, the width of the grid cells across the strips coincided with the width of a replication (36.6 m), and the length was also arbitrarily defined as 36.6 m (0.134-ha cells). The soil cores for each composite sample were collected following a random pattern from areas approximately 100-m^2 in size at the center of each cell. For second- or third-year trials soil samples were collected in the same manner but only from cells of the strips that received no K fertilizer. In this case, the cell length also was 36.6 m but the width was 18.3 m (the width of a treatment strip) and the cell area was (0.067 ha).

The soil samples were stored at 5 °C from 2 to 10 weeks after sampling, sieved through a 5-mm mesh, mixed thoroughly, and divided in two sub-samples. One set of samples from each site were maintained with the field moisture content while the other set was dried at 40 °C and crushed to pass through a 2-mm sieve. All soil analyses described in this section were done on duplicate soil samples. Soil moisture content of the field-moist samples was determined by drying a small portion to constant weight at 40 °C. Soil-test K was determined in the moist samples (MK) with the same 1*M* NH_4OAc procedure suggested for the North-Central Region by the NCR-13 committee (Warncke and Brown, 1998), except that to minimize sub-sampling error, the moist sample size was increased to twice the equivalent dry weight but the soil/solution ratio was maintained by using twice the amount of extractive solution. The set of oven-dried samples were used to extract K by three methods. One method (DK) was the standard 1*M* NH_4OAc procedure recommended for dried samples

(Warncke and Brown, 1998). To study the effects of re-wetting dried samples on extracted K (K reversion) two different methods described by Scott and Bates (1967) and Bates and Scott (1969) were used to pre-treat the sample before a standard 1*M* NH_4OAc extraction. One method (Rewet-K) consisted of adding of 1.4 mL of water to 2g of dried soil, allow samples to incubate for 24h at 25°C, extract them with 20 mL neutral 1 *M* NH_4OAc by shaking at 200 oscillations min^{-1} for 5 min, and measuring extracted K by atomic absorption spectroscopy. The second method (Octanol-K) was similar to the first one except for adding 1.4mL of water and 1.4 mL of n-octanol to the dried samples allowed to incubate for 24h at 40°C. Sample size was decreased to 2.0g from Scott and Bates original method to fit the laboratory condition while the soil/solution ratio was maintained. The length of time and temperature of the incubation were also changed slightly to utilize laboratory equipment and meet custom requirements for reporting results.

Composite samples from oven-dried soil samples collected the first year of the field trials were prepared in the laboratory to represent each predominant soil series in each field and were used to measure other soil properties. The cation exchange capacity (CEC) of the soils was estimated by summation of exchangeable Ca, Mg, K, Na, and neutralizable soil acidity (Warncke and Brown, 1998). Soil pH was measured using 1:1 soil/water ratio. Soil organic C was measured following the combustion method described by Wang and Anderson (1998) using a LECO CHN-2000 analyzer (LECO Corp., St. Joseph, MI). Soil texture was measured at the University of Nebraska Soil and Plant Analysis Laboratory by a method described by Kettler et al. (2001).

Grain yield was measured using combines equipped with commercial impact flow-rate yield monitors, moisture sensors, and global positioning system (GPS) receivers following a methodology used and described before in detail for evaluating various treatments (Bermudez and Mallarino, 2002; Wittry and Mallarino, 2004). Briefly, grain yield monitor data recorded every 1 s from experimental areas at least 40 m away from any field border, and any data point affected by common yield monitor errors (such as effects of waterways and unexpected combine stops) was deleted. Also, only data from combine passes from the center of each treatment strip (unaffected by treatment borders) were kept (for corn two passes 4.5 or 6 m wide each and for soybean either two passes 4.5 to 7.5 m wide each or one pass 9 m wide).

Geo-referenced yield monitor data recorded every second, soil-test K results for each sample, and digitized soil series maps (scale 1:12000, Iowa Cooperative Soil Survey, 2003) were imported into ArcView GIS software for appropriate spatial joining of the information based on polygons representing the experimental field layout. In fields where the experimental area included one soil series (or small areas of other soils did not encompass at least two replications of the experimental design) ArcGIS was used to calculate average soil-test K values and yields for each treatment and replication combination for the entire experimental area. In fields with two or more predominant soil series, calculated average soil-test K and yield data for each treatment and replication were within each soil. In the latter instances, minor areas of poorly represented soils were not used for this study. Data in Table 1 lists the 19 Iowa soil series represented in the study (the same soil series were present in several fields). For the purpose of the presentation of data and discussion of

results, each replicate within a field having a single soil series and each soil series from replicates with more than one soil is referred to as a "site". The study included 200 sites. . Each site and year combination is referred to as a site-year. The study included 200 site-years, 120 for corn and 80 for soybean.

The grain yield data used in this study is expressed as relative responses to K fertilization. Relative response was calculated for each replication site-year (as defined above) by dividing the yield of the control by the mean of the fertilized treatment and multiplying the result by 100. Each of these relative yield values was matched to an average soil-test K value that represented each replication and site-year combination. For example, the set of relative yield and soil-test data for a site-year having only one dominant soil included one pair of values (one soil-test K value and one relative yield value) for each field replication while for a site-year with two soil series, the set included one pair for each replication and soil series. These soil-test K values from each analysis method are represented by one point in figures showing relationships between K extracted by the different methods across all site-years. These pairs of soil-test K and relative yield values are represented by one point in figures relating relative yield to soil-test K for each analysis method across all site-years.

Relationships between amounts of K extracted by different methods were described and analyzed by linear or non-linear regression using the REG or NLIN procedures of SAS (SAS Institute, 2000). Critical soil-test K concentrations were determined by the statistical Cate-Nelson (CN) statistical method (Cate and Nelson, 1971) and by both the linear-plateau (LP) and quadratic-plateau (QP) segmented polynomial models (Waugh et al., 1973). The

critical concentration defined by the CN method was determined with the General Linear Models (GLM) procedure of SAS (SAS Inst., 2000) as the value that split the yield response data into the two groups that accounted for the largest proportion of the total variability (R^2). Critical concentrations defined by the segmented models were determined with the Nonlinear Model (NLIN) procedure of SAS, and represent the soil-test values at which the two portions of each model joined. We present models and critical concentrations for the best-fitting model for each crop and soil-test method. The best model was chosen as it has been suggested before (Dahnke at al., 1990; Mallarino and Blackmer, 1992) on the basis of statistical significance (significant at least at $P \leq 0.05$), R^2 values, and most reasonable fit as determined by observation of plot distribution residuals.

RESULTS AND DISSCUSION

Amount of K extracted by the soil tests

Soil-test K concentrations ranged across all sites from 73 to 346 mg K kg^{-1} for DK, 66 to 311 mg K kg^{-1} for Rewet-K, 24 to 251 mg K kg^{-1} for Octanol-K, and 25 to 224 mg kg^{-1} for MK. According to previous Iowa research the K extracted by the MK method should be the best estimate of readily available K for crops and approximately estimates exchangeable K without the effect of sample drying. The K extracted by the DK method ranged from a 3% increase to a 302% increase compared with K extracted by the MK method, and there was a 122% average increase across all site-years. The K extracted by the Octanol-K method ranged from a 60% decrease to 250% increase compared with K extracted by the MK method, although there was a 56% increase on average across all site-years. The K extracted

by the Rewet-K method ranged from a 7% decrease to a 297% increase compared with K extracted by the MK method, and there was a107% increase on average.

Our results confirmed previous research results in that soil sample drying usually significantly increases K extracted with the NH_4OAc method. The data also showed that on average across soils, incubating dried soil samples in the lab with water reduced extracted K only slightly compared with values for dried samples but incubating dried samples with water plus octanol resulted in K concentrations intermediate between concentrations from field-moist and dried soil analyses. Previous research (Scott and Bates, 1969) showed that pre-treating samples with water and octanol consistently reduced extracted K compared with dried samples but did not reduce it as much as with analysis on field-moist samples. In our study, however, K concentrations for the Octanol-K method sometimes were as large as for Rewet-K and DK. The difference between the two studies may be explained by laboratory conditions influencing the effect of the octanol pre-treatment and different soil properties. Although our duplicate analysis did not show consistently higher variation for the Octanol-K method compared with other methods, Scott and Bates (1969) suggested that uneven oven-drying conditions increase variability for this method. Their study included only seven Iowa soils from four different soil types (Floyd, Fayette, Clyde and Carrington) and several soil types from other states with contrastingly different properties (such as K levels, texture, organic matter, and possibly mineralogy) that may have maximized differences between soils and methods. Our study included several Iowa soils (Table 1) of less contrasting differences. Data in Table 1 also shows soil properties for trials by soil type. The data indicates that no

soil properties had an effect on the amount of K extracted (data not shown). Data in Table 2 are averages of the extracted K from soil types that are represented by more than two fields.

The amount of K extracted by NH_4OAc is influenced by the interaction between soil properties and environmental conditions in the field or laboratory, such as wet-dry and freeze-thaw cycles that influence K fixation and release to or from non-exchangeable forms. Early research (Dowdy and Hutcheson, 1963) showed that the clay mineralogy of the soils is closely related with K release or fixation observed upon soil sample drying. McLean and Watson (1985) stated that if the soil is relatively low in exchangeable K, soil sample drying causes scrolling of layers of the micaceous clay that releases non-exchangeable K but if the soil is high in exchangeable K or has had K added to it recently, drying usually drives water out from between the layers, causing them to collapse and trap K in a non-exchangeable form. Depending on the relative magnitude of these two mechanisms, a net release, fixation, or no change in exchangeable K could be observed. An additional factor that could be affecting K dynamic in soils and impacts of drying and wetting involves changes in the redox potential that affect the oxidation/reduction status of Fe in the soil. Conditions that favor low redox potential (such as high moisture and clay content) favor reduced forms of Fe which results in a stronger retention of K ions in interlayer positions of clays through various mechanisms (Khaled and Stucki, 1991; Shen and Stucki, 1994). This effect has not been well documented for soils, except those with moderate to high organic matter levels (Schindler et al., 2003). High organic matter has the potential to block access of K ions to interlayer sites favoring retention of K at less energetic exchange sites, but Iowa soils tend to

possess moderate to high organic matter, are moderately to poorly drained, and fine textured with higher CEC.

Figures 1 through 3 show relationships between K extracted by the four different methods used across all site-years of the study. Figure 1 shows that K extracted by DK and MK methods were linearly related across the range of values observed in the study (r^2 = 0.53), which indicated that the level of extractable K had no effect on the increase in extracted K by drying the soil. Data in Fig. 2 shows that Rewet-K also was linearly related to MK, with a slightly higher r^2 value of 0.59. However, data in Fig. 3 shows that although Octanol-K also was linearly related to MK the variation in the relationship (r^2 = 0.35) was much larger than for relationships between DK or Rewet-K with MK.

Data in Figs. 4 and 5 show that the relative difference between DK, Octanol-K, Rewet-K and MK tests (their ratio) and also the absolute difference decreased with increasing MK values. Because of the way differences were expressed, however, the relationships were exponential decreasing to a minimum for the relative differences (Fig 4) but linear for the absolute differences (Fig. 5). Moreover, the strength of the relationship was weaker for the absolute differences (Fig. 5 a,b, and c) than for the relative differences. Haby et al. (1988) and Barbagalata et al. (2005) also found that the increase in exchangeable K upon sample drying becomes proportionally less as the K level in the field-moist samples increased. Moreover, Haby et al. (1988) reported that at certain intermediate soil K content, K extracted from oven-dried soils equaled K extracted from field-moist samples (equilibrium concentration of K), but at higher K levels drying decreased extracted K compared with

field-moist samples. This extreme was not observed for Iowa soils in the study by Barbagelata el al. (2005) and in this study.

The additional K measured by DK, Rewet-K, or Octanol-K compared with MK was not correlated with soil CEC when it was expressed either as relative (DK/MK extraction ratio) or absolute differences (Figs. 6 and 7). The CEC of a soil depends mainly on the concentrations of clay and organic matter and clay mineralogy. Barber et al. (1961) found that soil texture (influencing CEC) and the level of exchangeable K in the soil were the primary factors influencing the change in exchangeable K measured upon sample drying. Interpretations of cause and effect concerning the lack of relationship between K tests differences and CEC in our study is difficult and speculation is risky. However, a possible explanation is the narrow range in texture and CEC (15 to 33 cmol kg^{-1}).

Field Correlation of yield response to K fertilization

Field response trials that encompass a wide range of soil-test levels and crop production conditions offer an appropriate basis for soil-test correlation and calibration. The wide range of growing conditions evaluated in this study resulted in a wide range of soil-test K values and grain yield response to K fertilization. Relative yield, which is an expression of yield response, varied from 78 to 109% across all site-years. Relationships between relative corn and soybean yield and soil K measured with the DK and MK tests are shown in Figs. 8 and 9. The best fit models for each crop and K test indicate a poor capacity of both DK and MK tests to predict a crop response to K fertilization. This variability should not be

explained mainly by experimental error because we included three to six replications in the trials and the laboratory analyses were done in duplicate. However, only in a few instances the variation in the relationship could be explained by environmental factors such as very low yield levels due to deficient or excessive soil moisture. Previous research in Iowa and other states (Beegle and Oravec, 1990; Mallarino and Blackmer, 1994) also showed poor relationships between corn relative yield and soil K extracted with NH_4OAc from dry samples. A rather similar result for the DK tests also was observed in recent Iowa research (Barbagelata et al., 2005) that included almost twice the number of sites, but the predictive capacity of the MK test was significantly better than in our study. We believe that differences for MK between the studies result from the fewer number of sites and the samples we selected for our study.

It is important to emphasize that the response models shown as well as the calculated estimated critical K concentrations should be used only for comparison of the four tests we used in this study and not to indicate critical levels that should be used in production agriculture. The much larger data set used by Barbagalata et al. (2005) and additional ongoing research by Dr. A. Mallarino's research group will be used for that purpose. For example, Figs. 8 and 9 suggest that critical K concentrations are lower for soybean than for corn, but this was not the case in the study by Barbagelata et al (2005) and more recent unpublished research. Critical concentrations for DK (Fig. 8) were 220 mg kg^{-1} and 158 mg kg^{-1} for corn and soybean, respectively. Critical concentrations for MK (Fig. 9) were 160 mg kg^{-1} and 70 mg kg^{-1} for corn and soybean, respectively. Figure 10 show relationships between relative crop yield and soil K measured with the Rewet-K method. The strength of

the relationship for corn was approximately similar to those for DK and MK methods. This result indicates that, although the determined critical concentrations differed, these three methods would be approximately similar concerning their value to predict corn response to K fertilization. The relationship between relative soybean yield and Rewet-K (Fig. 10) was slightly better than for corn with this method and for both crops with the DK method (Fig. 8), but was worse than for the MK method (Fig. 9). We believe this was the result of the sample set used in this experiment, because no obvious reason can explain that some soil test K methods are better for corn and others better for soybean.

Figure 11 show relationships between relative crop yield and soil K measured with the Octanol-K method. Soil K measured with this method related to yield response in a very different way than the other three methods and it did not predict well crop response to K fertilization. There was a linear fit for corn and no evidence of a critical K concentration within the range of observations, and there was no significant relationship for soybean. Obviously these results for the Octanol-K test cannot be explained by limitations of the sample set used and indicate that this test is not good to predict crop response to K fertilization in Iowa.

CONCLUSIONS

The amount of soil K measured with the DK test, which is the K test being used in Iowa and most of the world, was significantly higher than amounts measured by the MK test. The two pre-treatments for dried samples, rewetting with water and the addition of n-octanol,

extracted less K than the DK test in most soils, and the differences between the amount of K extracted was not clearly related to any measured soil property. There was only a weak relationship with CEC for the difference tests. The difference between DK and MK, Rewet K, and MK where larger as the CEC was greater, Octanol and MK was less as the CEC was higher.

The results showed that treating dried soil samples with octanol did not measure plant-available appropriately and was the worst method concerning prediction of crop response to K fertilization. Treating dried samples with water, however, resulted in a K test that was approximately similar to DK and MK concerning prediction of crop response to K fertilization. The Rewet-K test would be more practical for routine testing than the MK test, and should be preferred if its capacity to predict crop response to K is equal or better the MK test. Our results suggested, however, that Rewet-K is not clearly better than the commonly used DK test and that its use for routine testing would not be justified. However, because the number of sites and samples in this study was limited, further field calibration research with the Rewet-K test is justified.

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Table 1. Information on soils and soil proprieties for trials evaluating the response of corn and soybean to K fertilization.

Series	Subgroup	Family	Subsoil K†	Texture‡	Clay %	CEC	pH	OM%
Calco	Cumulic Endoaquolls	Fine-silty, mixed (calcareous), mesic	VL+	SICL	19.25	30.64	7.74	4.98
Canisteo	Typic Endoaquolls	Fine-loamy, mixed (calcareous), mesic	VL-	CL	19.77	26.12	6.62	5.53
Clarion	Typic Hapludolls	Fine-loamy, mixed, mesic	VL+	L	20.37	26.37	6.44	5.17
Clyde-Floyd	Typic Endoaquolls	Fine-loamy, mixed, mesic	VL-	CL	11.70	15.27	6.13	3.77
Colo-Ely	Cumulic Endoaquolls	Fine-silty, mixed, mesic	VL-	SICL	20.83	19.67	6.92	3.30
Crippin	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL-	L	22.25	20.62	6.44	4.68
Kenyon	Typic Hapludolls	Fine-loamy, mixed, superactive, mesic	VL-	L	17.40	17.08	5.84	4.27
Killduff	Dystric Eutrudepts	Fine-silty, mixed, mesic	VL+	SICL	18.90	20.71	6.96	3.32
Lawler	Aquic Hapludolls	Fine-loamy over sandy or sandy-skeletal, mixed, mesic	VL+	L	16.83	24.69	5.84	4.12
Mahaska	Aquertic Argiudolls	Fine-montmorillonitic, mesic	L	SICL	16.35	22.43	7.02	4.35
Nevin	Aquic Argiudolls	Fine-silty, mixed, mesic	L	SICL	15.95	16.11	5.75	3.10
Nicollet	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL+	L	21.41	26.16	6.53	4.93
Readlyn	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL+	L	13.05	18.41	5.82	4.23
Saude	Typic Hapludolls	Coarse-loamy over sandy or sandy-skeletal mixed, mesic	VL+	L	11.90	20.47	5.84	3.61
Spillville	Cumulic Hapludolls	Fine-loamy, mixed, mesic	VL+	L	17.77	25.67	6.73	4.68
Taintor	Vertic Argiaquolls	Fine-montmorillonitic, mesic	VL+	SICL	15.85	22.86	7.34	3.93
Tama	Typic Argiudolls	Fine-silty, mixed, mesic	VL+	SICL	18.48	20.79	6.70	4.10
Webster	Typic Endoaquolls	Fine-loamy, mixed, mesic	VL-	CL	19.04	25.44	6.59	5.14
Wiota	Typic Argiudolls	Fine-silty, mixed, mesic	VL+	SIL	N/A	N/A	N/A	N/A

† Subsoil K (30-60 cm depth). Very Low minus (VL-) <25 mg K kg^{-1}, Very Low plus (VL+) 25-50 mg K kg^{-1}, Low (L) 50-79 mg K kg^{-1} (ISPAID, 2004).

‡ Texture of the surface horizon, CL= Clay loam, FSL= Fine sandy loam, L= Loam, SICL= Silty clay loam, SIL= Silty loam, SL= Sandy loam (ISPAID, 2004).

Table 2. Information on average K extracted for soil types represented in at least two fields.

Series	Subgroup	Family	Subsoil K†	Texture‡	Moist	Octanol	Rewet	Dry
Calco	Cumulic Endoaquolls	Fine-silty, mixed (calcareous), mesic	VL+	SICL	85	63	152	162
Canisteo	Typic Endoaquolls	Fine-loamy, mixed (calcareous), mesic	VL-	CL	65	93	138	152
Clarion	Typic Hapludolls	Fine-loamy, mixed, mesic	VL+	L	84	106	151	168
Colo-Ely	Cumulic Endoaquolls	Fine-silty, mixed, mesic	VL-	SICL	51	98	136	127
Killduff	Dystric Eutrudepts	Fine-silty, mixed, mesic	VL+	SICL	81	130	163	156
Mahaska	Aquertic Argiudolls	Fine-montmorillonitic, mesic	L	SICL	88	176	156	144
Nicollet	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL+	L	73	102	148	168
Readlyn	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL+	L	66	109	104	125
Saude	Typic Hapludolls	Coarse-loamy over sandy or sandy-skeletal mixed, mesic	VL+	L	96	139	127	134
Spillville	Cumulic Hapludolls	Fine-loamy, mixed, mesic	VL+	L	49	43	115	96
Tama	Typic Argiudolls	Fine-silty, mixed, mesic	VL+	SICL	101	156	167	166
Webster	Typic Endoaquolls	Fine-loamy, mixed, mesic	VL-	CL	85	106	140	154

† Subsoil K (30-60 cm depth). Very Low minus (VL-) <25 mg K kg^{-1}, Very Low plus (VL+) 25-50 mg K kg^{-1}, Low (L) 50-79 mg K kg^{-1} (ISPAID, 2004).

‡ Texture of the surface horizon, CL= Clay loam, FSL= Fine sandy loam, L= Loam, SICL= Silty clay loam, SIL= Silty loam, SL= Sandy loam (ISPAID, 2004).

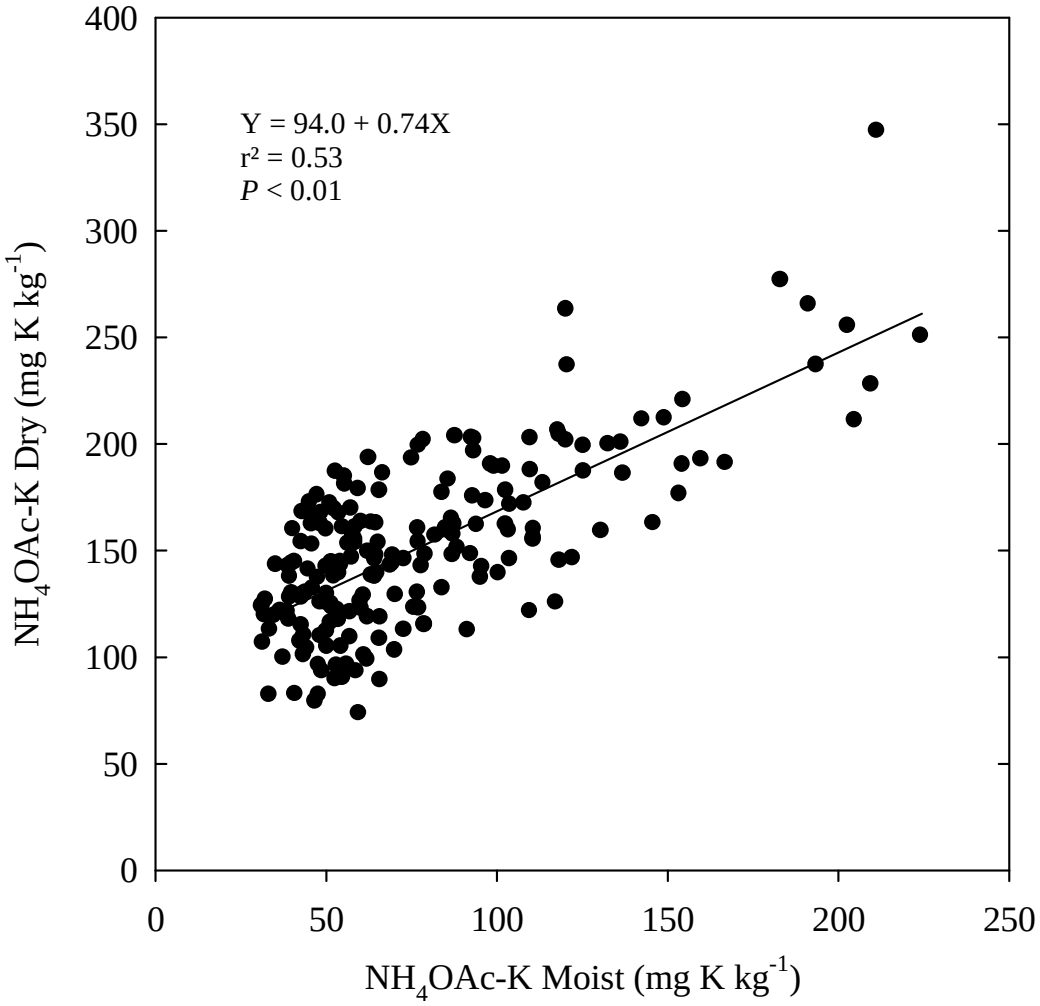

Figure 1. Relationship between exchangeable K measured with ammonium acetate (NH_4OAc) based on field-moist (Moist) and oven-dried (Dry) soil samples.

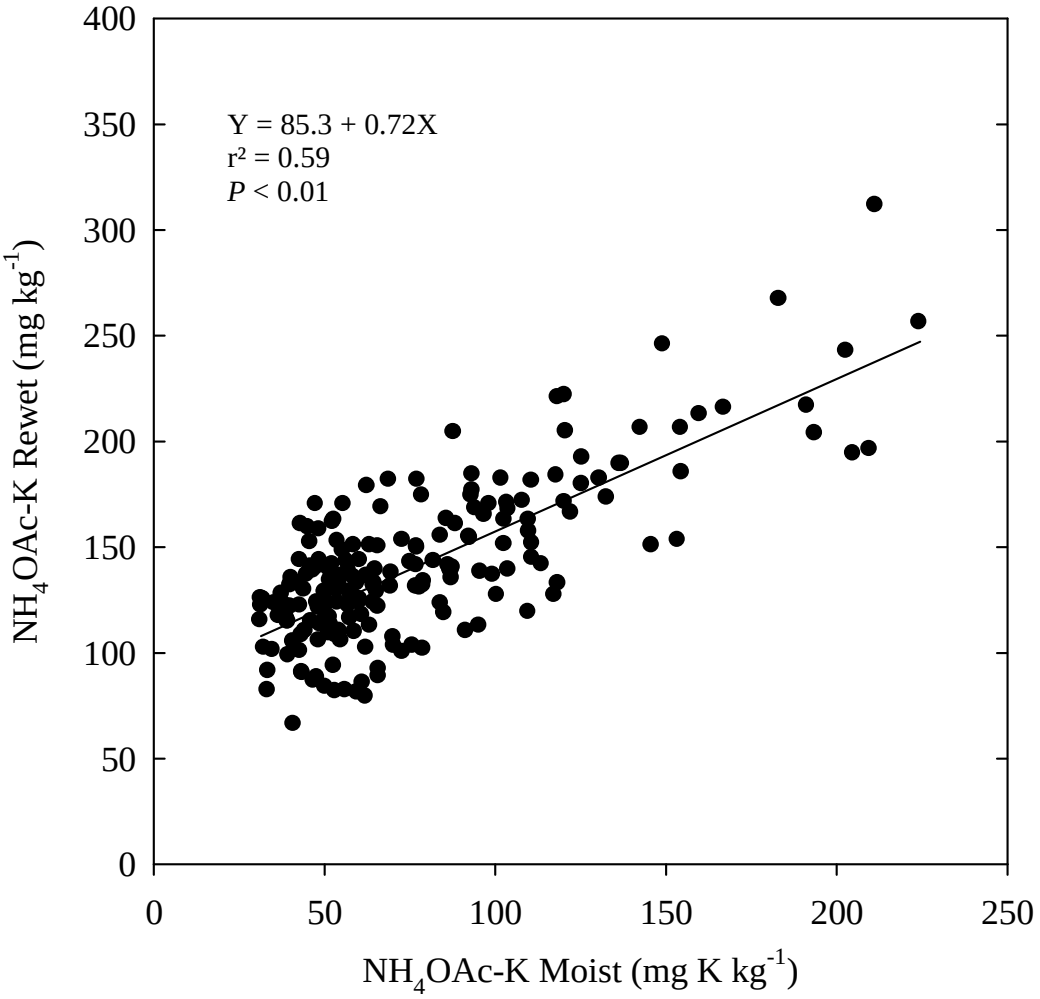

Figure 2. Relationship between exchangeable K measured with ammonium acetate (NH_4OAc) based on field-moist (Moist) and oven-dried rewet (Rewet) soil samples.

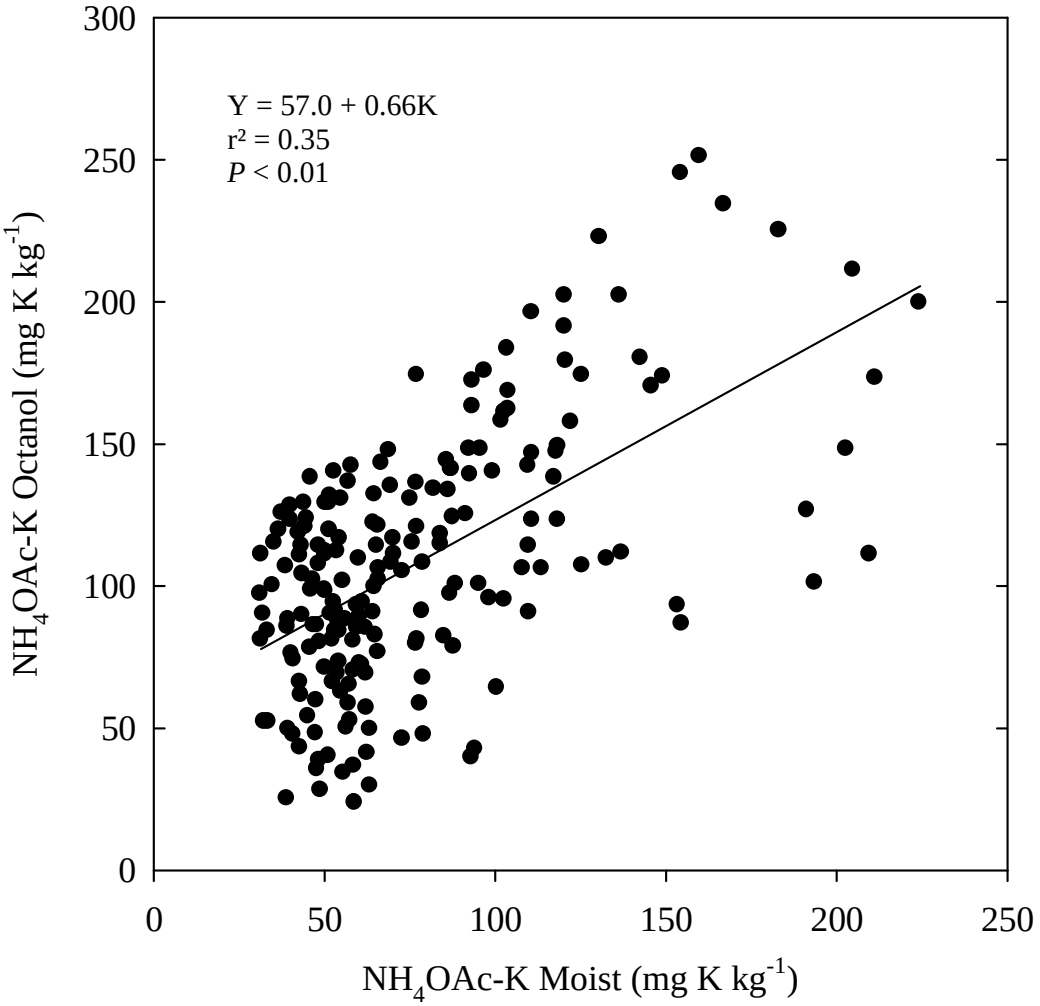

Figure 3. Relationship between exchangeable K measured with ammonium acetate (NH_4OAc) based on field-moist (Moist) and oven-dried n-octanol-treated (Octanol) soil samples.

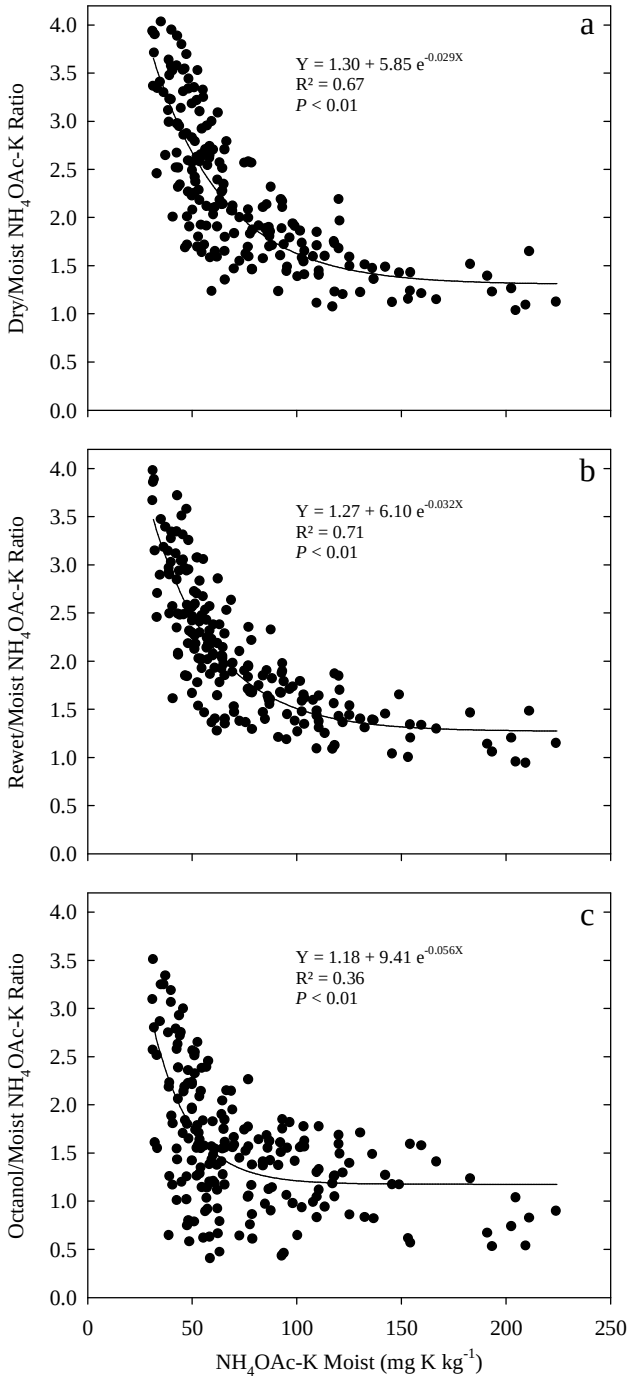

Figure 4. Relationships between exchangeable K measured with ammonium acetate (NH_4OAc) based on field-moist soil samples (Moist) and the relative difference from soil K measured with the same extractant but based on oven-dry soil samples (Dry) (a), oven-dry rewet soil samples (Rewet) (b), and oven-dry n-octanol treated soil samples (Octanol) (c).

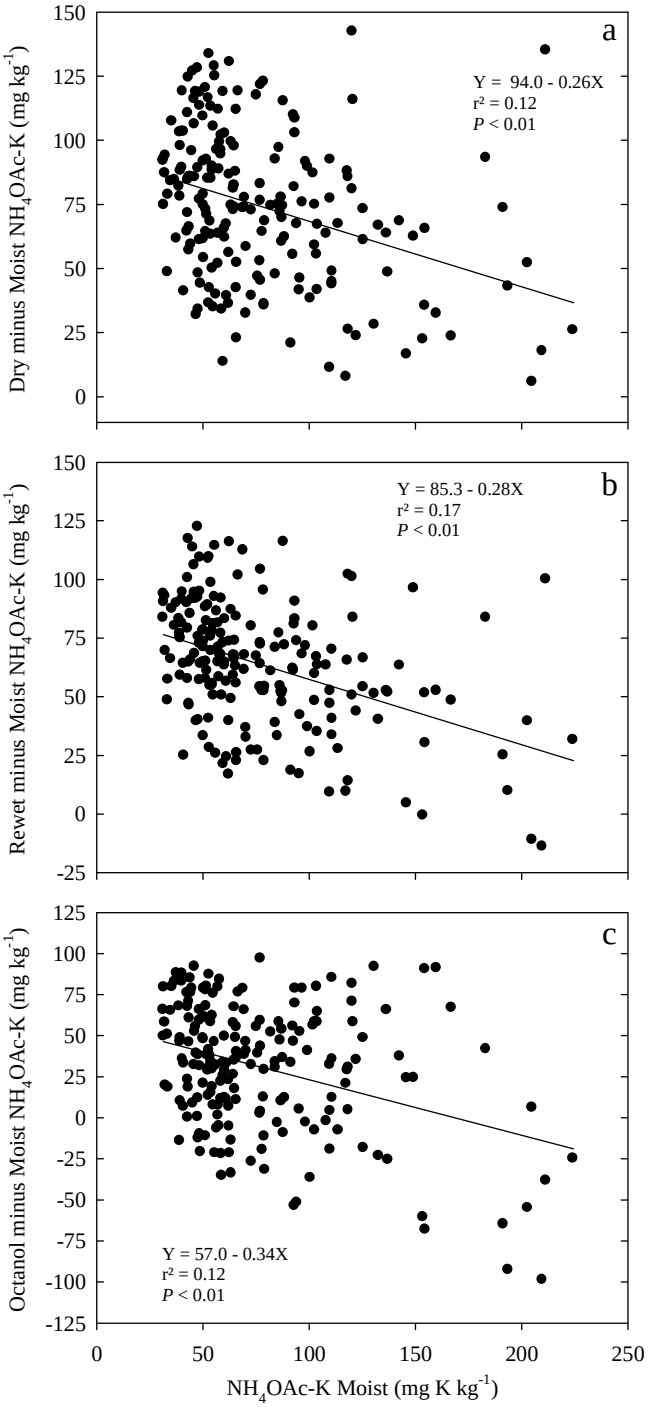

Figure 5. Relationships between exchangeable K measured with ammonium acetate (NH_4OAc) based on field-moist soil samples (Moist) and the absolute difference from soil K measured with the same extractant but based on oven-dry samples (Dry) (a), oven-dry rewet samples (Rewet) (b), and oven-dry n-octanol treated samples (Octanol) (c).

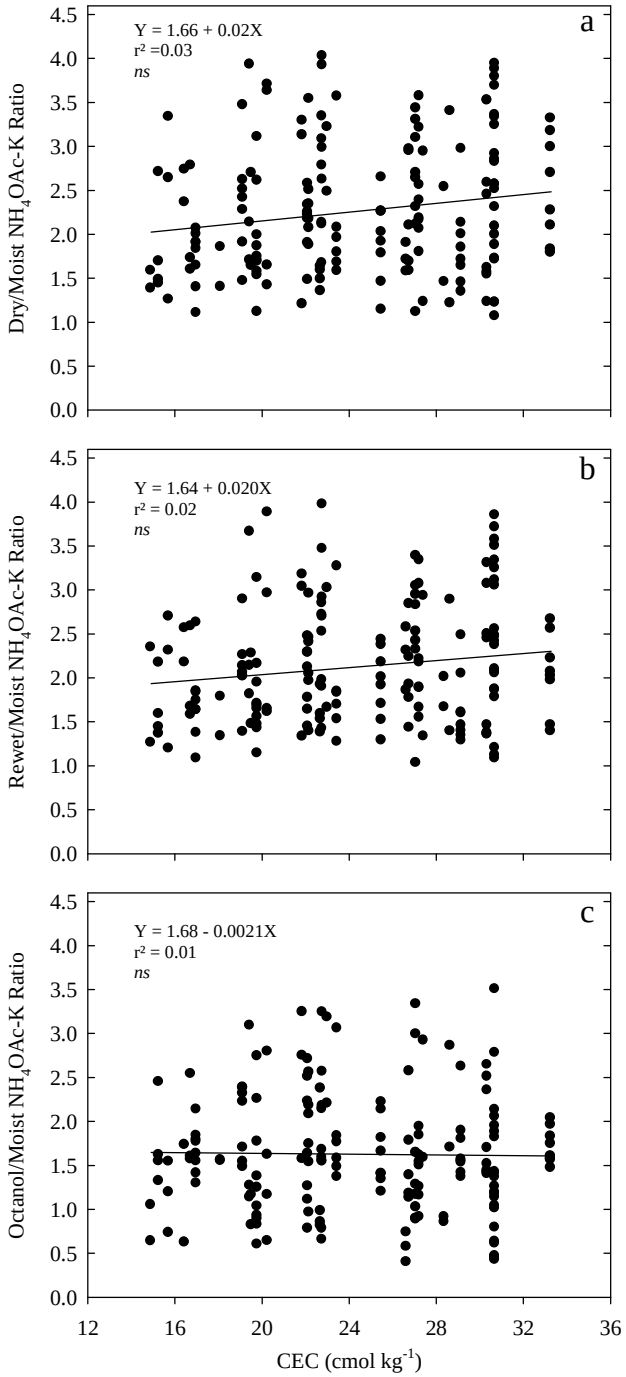

Figure 6. Relationships between cation exchange capacity (CEC) and the relative difference in soil K measured with ammonium acetate (NH_4OAc) based on field-moist soil samples (Moist) and oven-dried samples (Dry) (a), oven-dry rewet samples (Rewet) (b), or oven-dry n-octanol treated samples (Octanol) (c).

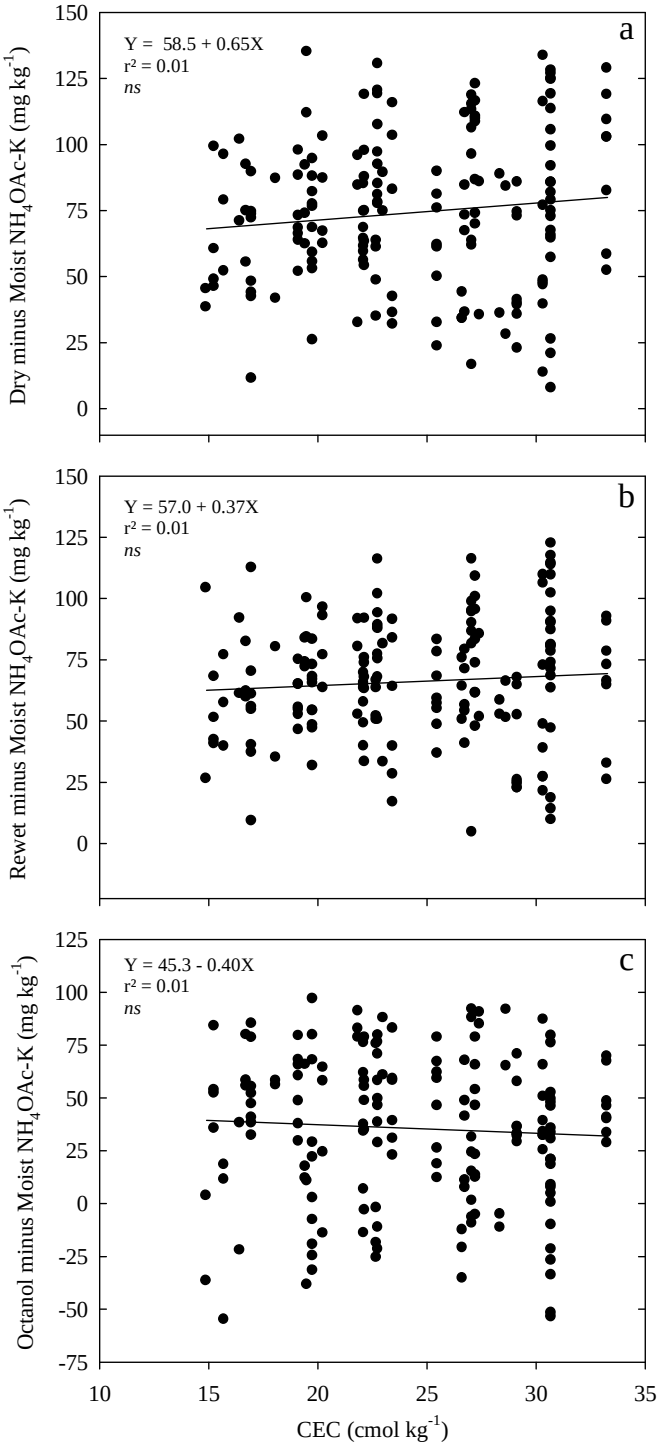

Figure 7. Relationships between cation exchange capacity (CEC) and the absolute difference in soil K measured with ammonium acetate (NH_4OAc) based on field-moist soil samples (Moist) and oven-dried samples (Dry) (a), oven-dry rewet samples (Rewet) (b), or oven-dry n-octanol treated samples (Octanol (c).

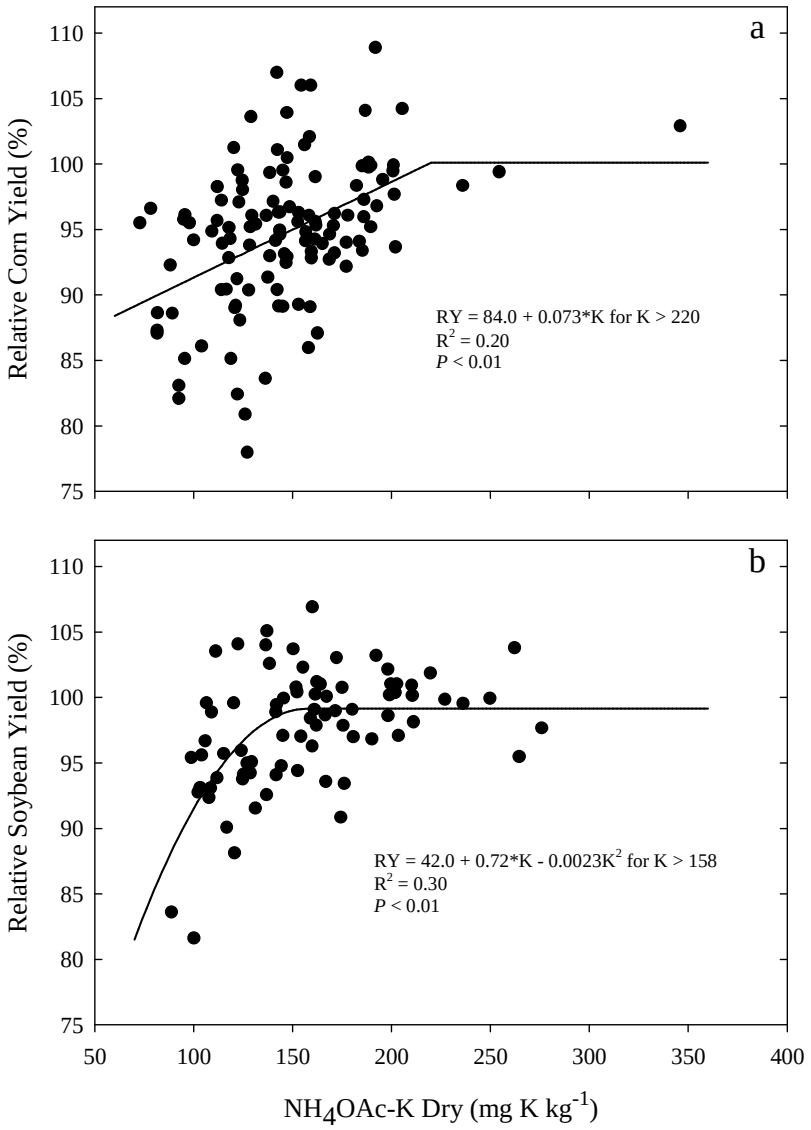

Figure 8. Relationships between relative corn yield and exchangeable K measured with ammonium acetate (NH_4OAc) based on oven-dry (Dry) soil samples, with linear-plateau model fitted to corn data points (a) and quadratic-plateau model fitted to soybean data points (b).

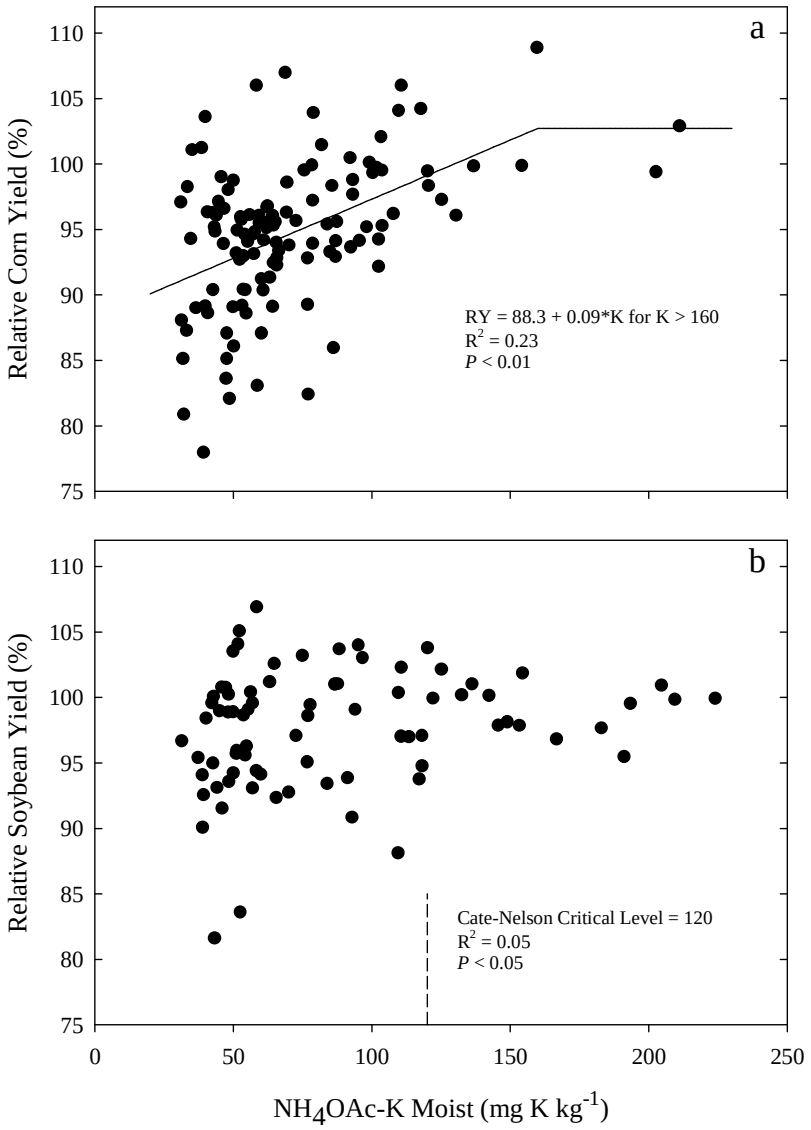

Figure 9. Relationships between relative corn yield and exchangeable K measured with ammonium acetate (NH_4OAc) based on field moist (Moist) soil samples, with linear-plateau model fitted to corn data points (a) and Cate-Nelson model fitted to soybean data points (b).

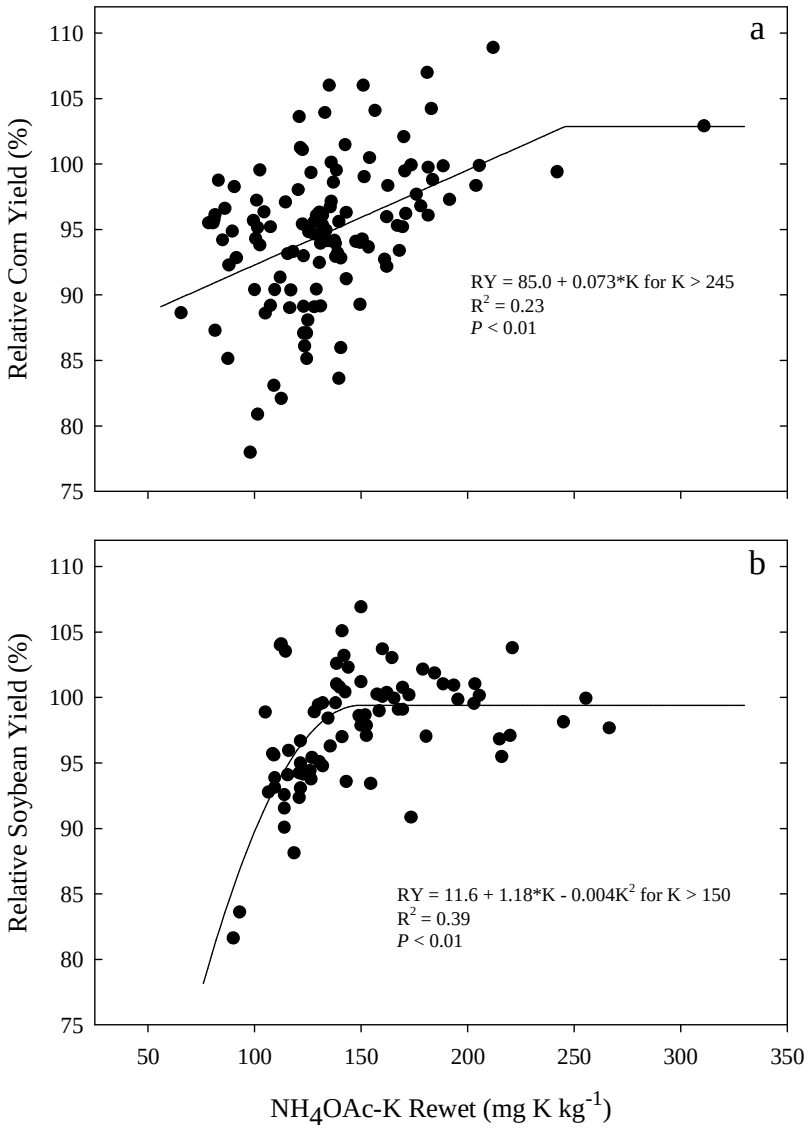

Figure 10. Relationships between relative corn yield and exchangeable K measured with ammonium acetate (NH_4OAc) based on oven-rewet-K (Rewet) soil samples, with linear-plateau model fitted to corn data points (a) and quadratic-plateau model fitted to soybean data points (b).

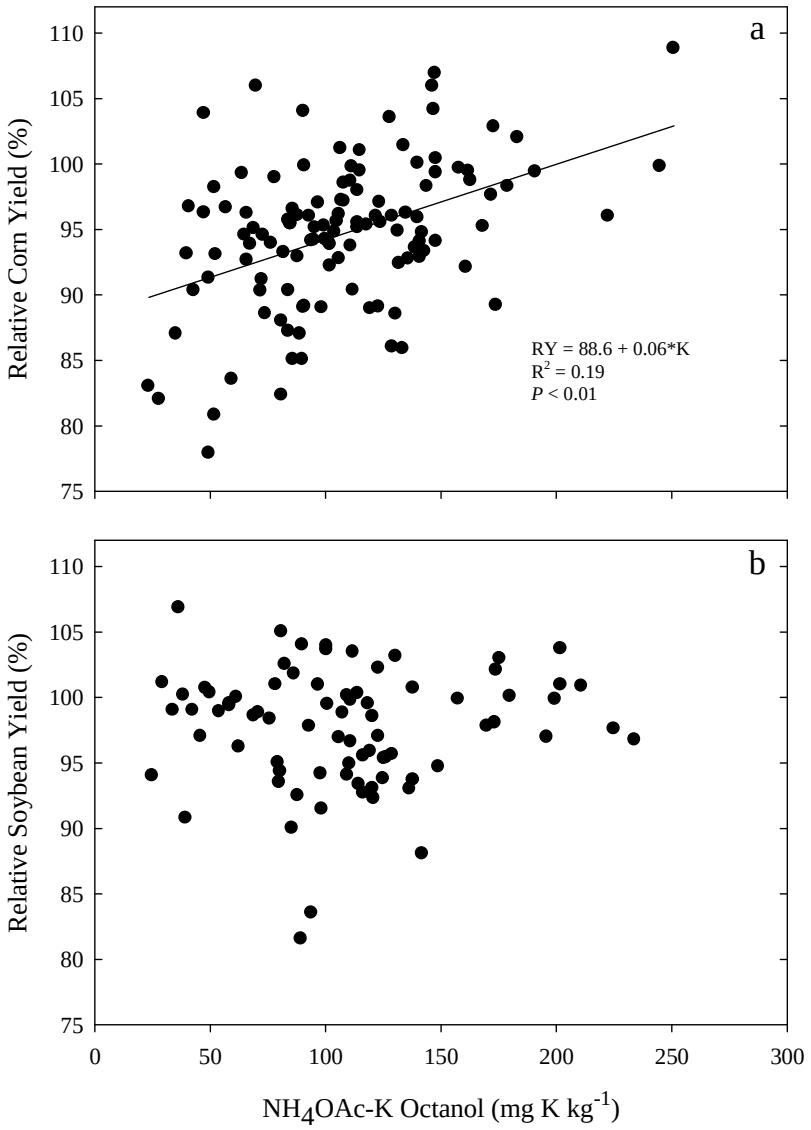

Figure 11. Relationships between relative corn yield and exchangeable K measured with ammonium acetate (NH_4OAc) based on oven-dried octanol-K (Octanol) soil samples, with linear-plateau model fitted to corn data points (a). No model fit soybean data significantly (b).

CHAPTER 3: GENEERAL CONCLUSIONS

The goal of this research was to expand recent work in Iowa by further studying potassium (K) extraction methods and its use to evaluate soil K sufficiency for crops. Specific objectives were to compare soil-test results and relationships between soil-test K and crop yield response to K fertilization when soil K is measured with the NH_4OAc extractant from field-moist and oven-dried soil samples and also after applying different treatments to oven-dried samples in an attempt to revert the effect drying has on the release of K.

The amount of soil K measured by drying soil samples (DK test), which is the K test being used in Iowa and most of the world, was significantly higher than amounts measured by extracting K form field-moist samples (MK test). The two pre-treatments for dried samples, rewetting with water (Rewet-K test) and the addition of n-octanol (Octanol-K test), extracted less K than the DK test in most soils, and the differences between the amounts of K extracted was not clearly related to any measured soil property. There was only a weak relationship with CEC for the difference tests. The difference between DK and MK, Rewet K, and MK where larger as the CEC was greater, Octanol and MK was less as the CEC was higher.

The results showed that treating dried soil samples with octanol did not measure plant-available K appropriately and was the worst method concerning prediction of crop response to K fertilization. Treating dried samples with water, however, resulted in a K test that was approximately similar to DK and MK concerning prediction of crop response to K fertilization. The Rewet-K test would be more practical for routine testing than the MK test, and should be preferred if its capacity to predict crop response to K is equal or better the MK

test. However, our results suggested that Rewet-K is not clearly better than the commonly used DK test and that its use for routine testing would not be justified because of the additional labor and time required. However, because the number of sites and samples in this study was limited, further field calibration research with the Rewet-K test is justified.

ACKNOWLEDGEMENTS

I would like to thank everyone involved in making my graduate project possible. First and foremost I would like to thank my Major Professor Antonio Mallarino for his wisdom, dedication, and indispensable aid in preparation of this manuscript. All the members of the ”Mallarino Soil Fertility Research Group”, all the staff from the Iowa State University Soil and Plant Analysis Laboratory and many other who helped me complete my research work. And to my wife Kelly and son Owen.

Thanks to all of you.